P. JÓŹWIK*, Z. BOJAR*

ANALYSIS OF GRAIN SIZE EFFECT ON TENSILE PROPERTIES OF Ni_3Al – BASED INTERMETALLIC STRIPS

ANALIZA WPŁYWU WIELKOŚCI ZIARNA NA PARAMETRY WYTRZYMAŁOŚCIOWE TAŚM ZE STOPU NA OSNOWIE FAZY MIĘDZYMETALICZNEJ Ni_3Al

The results of investigation of grain size effect on mechanical properties of Ni_3Al – based intermetallic strips measured in a tensile test at room temperature in air have been shown in the paper. Thin intermetallic strips with average grain size of 1, 5, 7, 26, 43 and 83 micrometers prepared by cold rolling and heat treatment at parameters chosen on the basis of our earlier completed recrystallization maps have been used for tensile tests.

It has been stated that increasing of basic γ' phase grain refinement caused intensive increase of tensile yield strength (TYS) and lower increase of ultimate tensile strength (UTS). The highest effectiveness of grain boundary strengthening has been found for average grain size up to 10 micrometers and was connected with a strong reduction of intermetallic material plasticity as well with increasing of its elasticity finding expression in TYS/UTS ratio. However, even for extremely fine-grained Ni_3Al sample (average grain size approximately 1 micrometer) a value of ultimate tensile elongation obtained at room temperature in air ("hard conditions" for intermetallic alloys sensitive to environmentally influenced brittleness) was still high – approximately 30% at tensile yield strength above 1200 MPa. The results obtained (for recrystallized Ni_3Al) were much better then those for appropriate alloys described in literature. In the analyzed grain size range, a classic exponential dependence of yield strength on average grain size was confirmed.

Tension test results were in good correlation with fracture structure. It has been stated that fractures of samples with different grain size were quite similar and mixture-type. There was cleavage fracture via micro-volume of each grain, related to ductile fracture via microregions along grain boundaries. For all tested range of grain sizes a fraction of ductile fracture microregions was high, but higher for coarse-grained samples with ultimate elongation ran into 70%.

A dependence of a phase structure (a fraction of γ disordered phase) and degree of γ' phase ordering on mechanical properties and fracture in a tensile test at room temperature in air was also discussed in the paper.

Keywords: nickel aluminide, thermomechanical processing, nanostructure, tensile properties, fracture

W pracy przedstawiono wyniki badań wpływu wielkości ziarna na parametry wytrzymałościowe taśm ze stopu na osnowie fazy międzymetalicznej Ni_3Al mierzone w próbie statycznego rozciągania, w temperaturze pokojowej, w atmosferze powietrza. Po szczegółowej analizie morfologii budowy ziarnowej materiału w stanie po wygrzewaniu rekrystalizującym prowadzonym przy wykorzystaniu map rekrystalizacji opracowanych we wcześniejszych badaniach do analizy wybrano próbki o średniej wielkość ziarna: 1 μm, 5 μm, 7 μm, 26 μm, 43 μm i 83 μm.

Stwierdzono, wraz ze wzrostem stopnia rozdrobnienia fazy γ' stanowiącej osnowę badanego materiału, intensywny przyrost granicy plastyczności oraz mniej intensywny przyrost doraźnej wytrzymałości na rozciąganie. Wykazano, że największa skuteczność umocnienia granicami ziaren (poprzez rozdrobnienie ziarna) ma miejsce w przedziale wielkości ziarna do ok. 10 μm, co odpowiada jednocześnie, zachodzącemu równolegle intensywnemu zmniejszeniu plastyczności materiału i zwiększeniu jego „wydolności sprężystej" wyrażonej stosunkiem R_e/R_m. Jednak nawet dla skrajnie drobnoziarnistego stopu (średnia średnica ziarna około 1 μm) wydłużenie uzyskiwane w próbie rozciągania prowadzonej w temperaturze otoczenia w powietrzu (bardzo ważne czynniki "obciążające" wyniki uzyskiwane dla próbek stopów intermetalicznych) jest bardzo wysokie, bo bliskie 30%, przy także wysokiej granicy plastyczności powyżej 1200 MPa. Uzyskane wyniki, dla stopu w stanie równowagowym (po rekrystalizacji), są nieporównywalnie korzystniejsze od odpowiadających im nielicznych danych dostępnych w literaturze.

Potwierdzono także, w rozpatrywanym przedziale wielkości ziarna, "klasyczny" przebieg wykładniczej zależności granicy plastyczności od wielkości ziarna. Wyniki badań statycznego rozciągania korespondują z budową fraktograficzną powierzchni przełomów. Stwierdzono, że przełomy badanego stopu w stanie bez umocnienia ale przy zróżnicowanej wielkości ziarna wykazują wiele wspólnych cech budowy – są efektem pękania łupliwego w mikroobszarach i pękania ciągliwego stref granic ziaren, natomiast różnią się udziałem obu tych składowych. Przełomy badanego stopu, w całym rozpatrywanym przedziale

* FACULTY OF ADVANCED TECHNOLOGY AND CHEMISTRY, MILITARY UNIVERSITY OF TECHNOLOGY, 00-908 WARSZAWA, 2 KALISKIEGO STR., POLAND

wielkości ziarna, charakteryzują się dużym udziałem odkształcenia plastycznego w granicach ziaren – zauważalnie większym dla ziarna o większych rozmiarach pozwalającym na uzyskanie wydłużenia równomiernego na poziomie 70%. Dyskutowano także problem wpływu budowy fazowej (udział nieuporządkowanej fazy γ) oraz zmian stopnia uporządkowania podstawowej fazy γ' na właściwości mechaniczne i budowę przełomów w próbkach po rozciąganiu w temperaturze pokojowej, w atmosferze powietrza.

1. Introduction

Alloys based on ordered intermetallic phase matrices are called to be up-to-date engineering materials with specific properties, potentially useful for various functional and structural applications [1–3]. Among them, intermetallic alloy based on the Ni_3Al phase show, as compared to currently used nickel superalloys, anomalous increase of yield point with temperature, good resistance to different forms of tribological wear and a perfect resistance to oxidation and other types of corrosion. They are mainly used for matrices, press tools, and furnace equipment used in thermo-chemical treatment, and – after directed crystallization – for turbine blades in jet engines [1–3, 6, 9].

In view of specific physico-chemical properties, a significant increase of these materials is expected in construction of small mechatronic systems (MECS type devices – Microtechnology-based Energy and Chemical Systems). If used as foils, they are or will be applied in the nearest future as elements for electromechanical micro-systems (heat exchangers, micro-reactors, catalysts, micro-servomotors, etc.), jet aircraft engines, car engines, or car catalysts [3–9].

In order to solve a problem of the production of Ni_3Al strips the international scientific group called the ***"Ni₃Al thin foils group"***, consisting of such well know centers like National Institute for Materials Science (Japan), Oak Ridge National Laboratory (USA), Max-Planck Institute fűr Eisenforschung (Germany), Oregon State University (USA), Korea Advanced Institute of Science and Technology (Korea) has been established [5] which testifies to the importance of the Ni_3Al strips problem.

2. Experimental procedure

Tests were carried out with the alloy based on the Ni3Al intermetallic phase (chemical composition: Ni-11.5Al-0.46Zr-0.02B (% wt.). As-cast samples have been worked out in a multistage process of heat treatment and plastic working (in strictly selected deformation and recrystallization conditions). A detailed analysis of the grain structure morphology of the material after recrystallization soaking, described earlier, [9], was applied for selection of samples with average grain size between 1.34 and 83 μm. The obtained results allowed the authors for plotting so called "recrystallization maps" taking into account mutual relations of primary technological parameters like cold work value and recrystallization temperature as well as material parameters like grain size and hardness. This procedure enabled precise conditions for plastic working and heat treatment processes parameters to be set in order to obtain materials with fixed structure and strengthening level.

The mictrustructural analysis was performed using analyzing scanning Philips XL30 LaB_6 and optical Neophot 2 microscopes. Tensile tests were performed in air at room temperature, at constant strain rate $10^{-3}s^{-1}$ using the Instron 8501 machine. Tensile specimens were cut from processed foils along rolling direction and then mechanically polished. The total length of the tensile specimens was 50 mm, the gauge length 20 mm and the gauge width 7 mm.

On the base of measurement results the yield point R_e, ultimate tensile strength, and permanent elongation after break were determined.

3. Results and discussion

The investigated alloy reveals a diphase structure. The main structural component (the matrix) is the ordered secondary solid solution γ' based on the intermetallic Ni_3Al phase, while the second one (regions with fine bright releases) – is a mixture of a γ' phase and a γ phase – disordered solid solution of aluminum in the nickel lattice – Fig. 1.

Investigations carried out on samples with 1, 5, 7, 26, 43 and 83 μm average size diameters were performed in order to determine the effectiveness of the size boundary strengthening for dislocation blocking and the effectiveness of the axial tension strengthening.

The investigations, carried out on sample inspection lots with size enabling statistical processing of the measurement results, showed a clear influence of a grain size on strengthening parameters measured in a static tensile test at room temperature and air atmosphere (a setting-up for recalculated charts of strengthening via tensile tests is shown in Fig. 2).

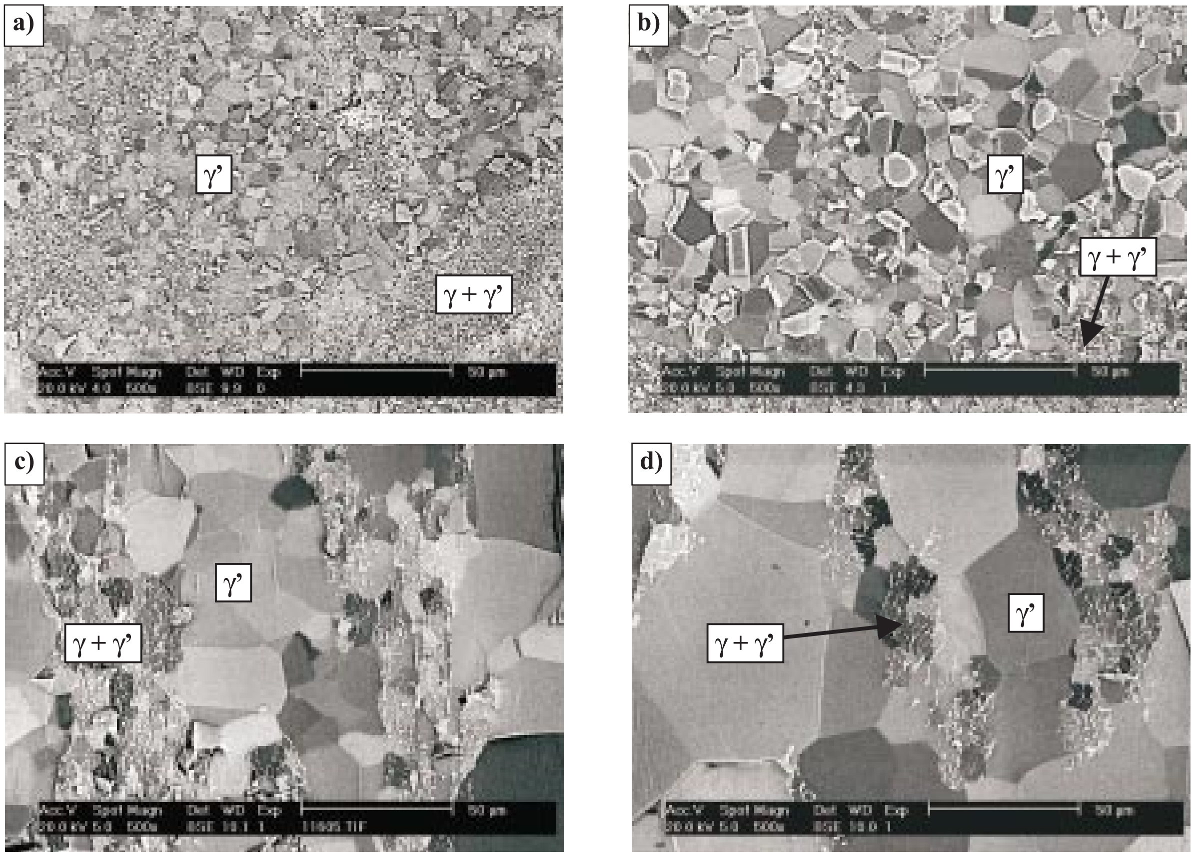

Fig. 1. Microstructure of the Ni_3Al – based alloy samples with average grain size: a) 5μm, b)7μm, c) 26 μm, and d) 43 μm

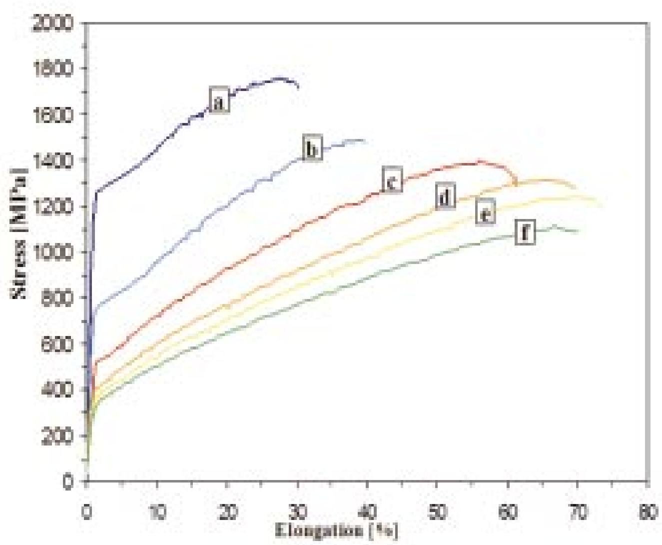

Fig. 2. Exemplary strengthening curves obtained for recrystallized samples of Ni_3Al (Zr, B) alloy with various grain size (description in Table 1) after static tensile testing at room temperature in air atmosphere

A significant increase of the yield point and a lower increase of the tensile strength was observed with an increase of a refinement of the γ' phase structure.

The best effectiveness of the grain size strengthening (via grain size reduction) is observed for the 10 μm grain size (Fig. 3) which corresponds to the parallel decrease of the yield point of the material and the simultaneous increase of "elasticity factor" expressed in R_e/R_m terms (Fig. 4). It is worth to notice, that even for ultra fine-grained alloy (with average grain diameter below

TABLE 1

A comparison of tensile properties for Ni_3Al (Zr, B) strips 300–500 μm thick obtained in static tensile tests at room temperature in air atmosphere

Material condition	γ'– phase equivalent grain diameter [mm]	Ultimate tensile strength [MPa]	Tensile yield strength [MPa]	Elongation [%]
variant **a**	1.34±0.5	1757±53	1254±31	28.5±6
variant **b**	4.8±2	1490±23	767±18	38±5
variant **c**	7.3±2.4	1396±19	560±6	58±9
variant **d**	26.2±10	1309±12	396±9	66.5±7
variant **e**	43.1±15	1237±14.5	360±8	71±8
variant **f**	83±19	1109±12	310±9	68±4
variant **g** ([3])	18	350	280	3
variant **h** ([3])	25	290	240	4
variant **i** ([3])	62	250	150	5

1.5 μm), elongation observed in tensile tests at room temperature in air (very important factors adversely influencing results for intermetallic alloy samples) is very high (up to 30%) and it is accompanied also by a very large yield point (above 1200 MPa). Our results obtained for alloy after recrystallization are significantly more advantageous as compared to presented in the literature [3].

In diagrams (Figs 3 and 4) results by M. Demura et al. [3] are plotted for comparison. They concern to thin strips of similar chemical composition and size (thickness 315 μm) to the Ni_3Al (Zr, B) strips investigated in the paper. There are no numerous results in the literature related to investigations of the Ni_3Al strips, these are works carried out by the so called the "Ni_3Al thin foils group" mentioned in the introduction. The Ni_3Al (Zr, B) alloy prepared by the authors is characterized by the 14 times larger permanent elongation, the five-fold tensile strength, and the 3.5-fold yield point as compared to the alloy tested in [3] (see Figs 2, 3, and the Table 1).

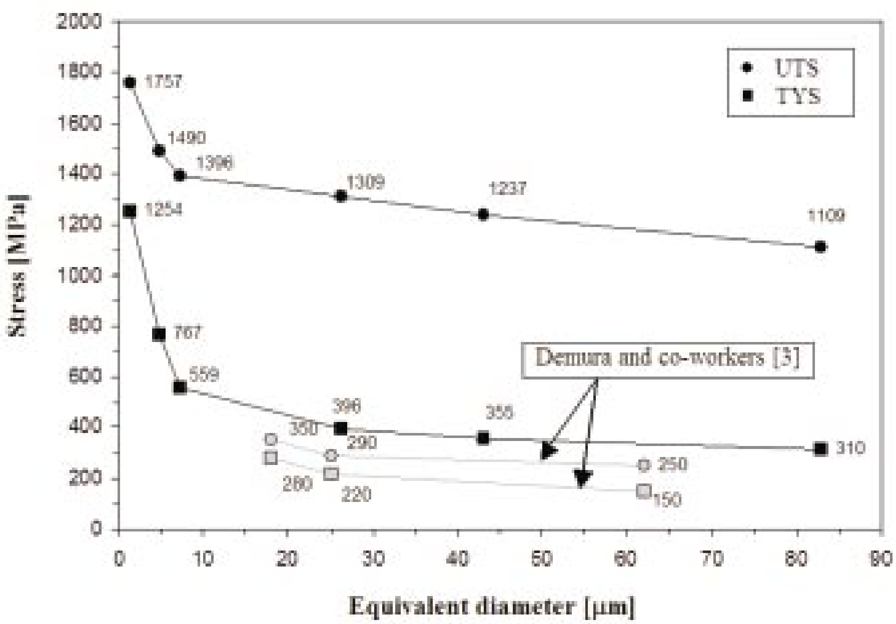

Fig. 3. Changes of tensile properties (TYS, UTS) of $Ni_3Al(Zr,B)$ alloy as a function of grain size (data from Tab. 1)

According to the Hall-Petch equation, a classic exponential dependence of yield point on grain size was found (results obtained for 6 grain size values). The exponent index is typically equal to −0.5, however E.M. Schulson and S. Hanada in papers [10, 11] used a value −0.8. Checking many various variants for the exponent

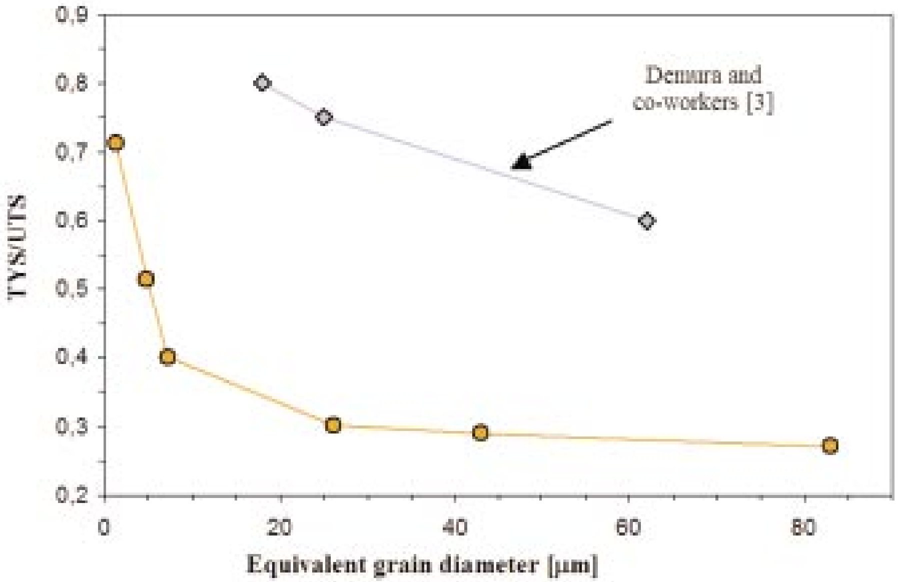

Fig. 4. The change of TYS/UTS ratio versus grain size of the Ni_3Al (Zr, B) alloy

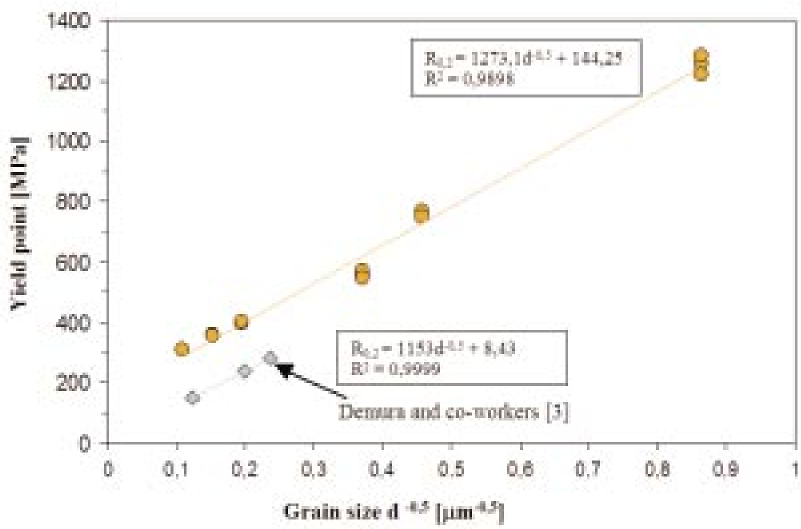

Fig. 5. The Hall-Petch relationship for: Ni_3Al (Zr, B) alloy investigated by authors (circle) and according to literature data (Ni-25Al [3])

index, the authors achieved the best fit to the Hall-Petch equation using typical value −0.5, the same was confirmed in the paper by M. Demura [3].

As far as the authors' and the M. Demura et. al., [3], experiments are concerned, an especially large difference can be attributed to the free term in the Hall-Petch equation: the value 144.25 in the present paper and the value 8.43 in the paper [3] adequately. This free term in the Hall-Petch expression is related to the lattice friction stress and makes dislocation motion drag depend directly on dislocation motion velocity, and indirectly on the number of active grains per alloy unit volume in the deformation process.

In the current experiment, however, a bit larger slope of the straight line (the smaller value of the directional coefficient) in the Hall-Petch equation, better approximating the authors' results, gives an evidence that the generation of dislocations (propagation of deformation from grain to grain) in the investigated alloy is more difficult.

The results of static tensile tests correspond to the structure of fracture surface. The investigated alloy samples in the whole grain size range can be characterized by high plastic deformation fraction in grain boundaries – apparently higher for larger grains which makes relative elongation at the 70% level possible.

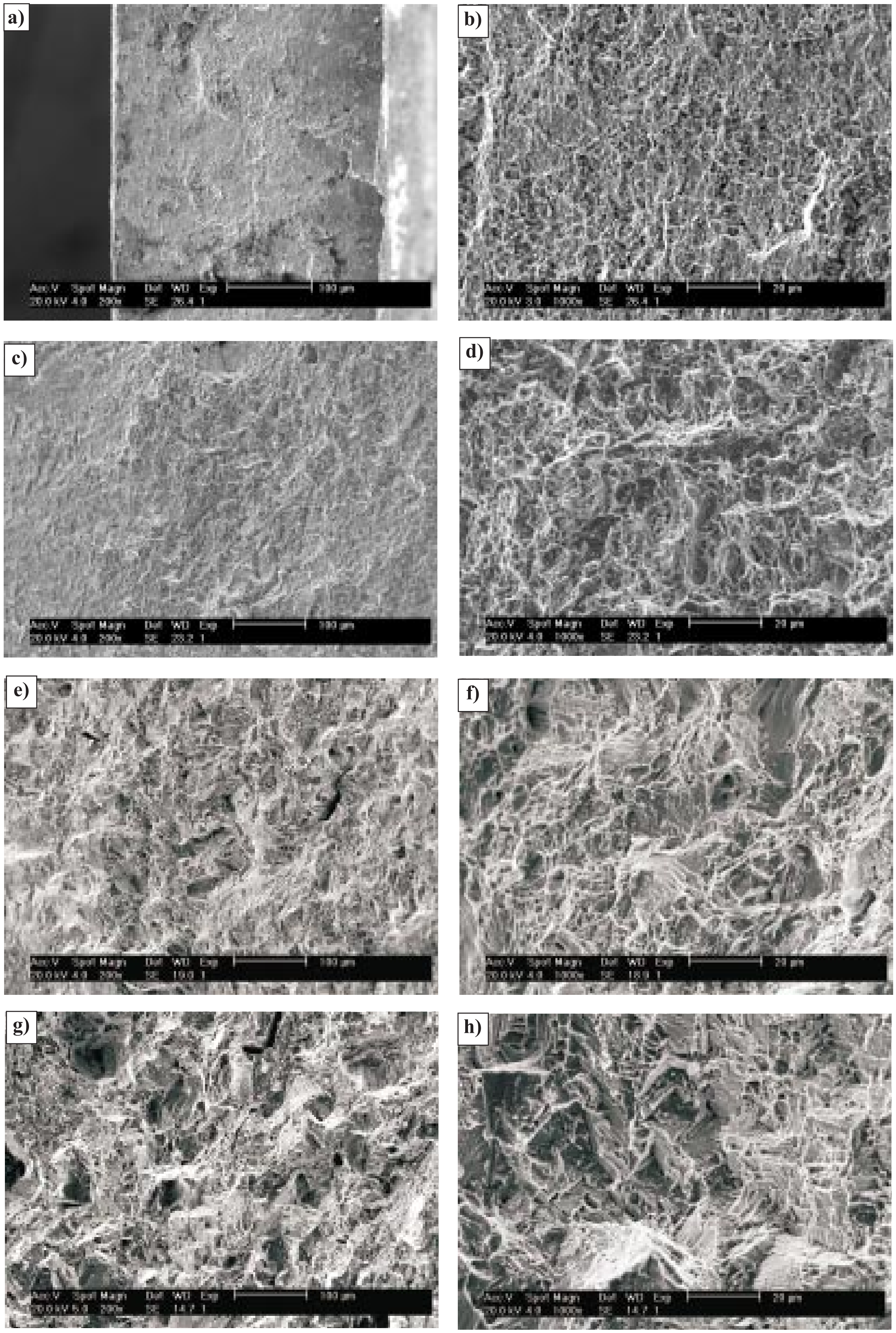

Fig. 6. The structure of fractures after static tensile tests of the Ni_3Al (Zr, B) alloy at room temperature in air: a, b) variant a (1.34 μm); c, d) variant c (43.1 μm); e, f) variant d (83.4 μm) (description in Tab.1)

It was stated that fractures of the investigated alloy, despite the diversified grain size, exhibit many similar structural properties – they are effect of fissile cracking in micro-zones (limited to single grain dimensions) and ductile cracking of the grain boundary zones (corresponding to the brittle destruction of a grain volume), but they differ in relative fraction of these components. The above analysis, however, if not related with other structural changes, could lead to inconsistency with observed dependence of elongation increase on enlargement of an average grain size (in this situation a relative enlargement of areas inclined to fissile cracking occurs which means that permanent elongation should be more and more lower). One should take into account, however, a competitive effect of an influence of the structure homogenizing process (decay of ($\gamma + \gamma'$) regions occurring simultaneously with recrystallization process. On the other hand, for ultra fine-grained alloys (grain size below 10 μm) the elongation is relatively lower than that resulting only from cracking mechanism (in this case a fraction of ductile cracking in a zone of developed surfaces of grain boundaries is large). This is caused by a competitive phenomenon of particularly high alloy strengthening for this grain size range during tensile test (Fig. 2). It was stated that for alloys with the largest grain diameter (83 μm after long-term recrystallization) the trend of elongation increase with the grain size enlargement was reversed, since a homogeneous material cracks mainly by separation at fissile planes. Nevertheless, the obtained fissile fracture is highly developed ("three-dimensional" – Fig. 6 e, f) and resulted elongation is still extremely high (68%).

4. Conclusions

It was stated on the base of obtained results that the grain size (expressed in equivalent diameter) significantly influences the investigated material strengthening parameters measured in static tensile test at room temperature in air atmosphere. For grains below 10 μm a large increase of yield point and a bit lower increase of the tensile strength were observed. This influence is much lower for large grains.

For extremely fine-grained condition the obtained yield point and tensile strength values were a few times better than reported in the literature for chemically similar alloys: 14-times higher permanent elongation, 5-fold larger tensile strength and the 3.5-fold yield point.

The "classic" Hall-Petch exponential dependence of yield point on grain size was confirmed for tested grain size range, too.

The results of statitc tensile tests correspond to a fractural structure of fracture surface. The fractures of the investigated alloys, in the entire grain size range, are characterized by a large plastic deformation within grain boundaries – noticeably higher for larger grains which allows for an uniform elongation at the 70% level.

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REFERENCES

[1] J. Bystrzycki, R.A. Varin, Z. Bojar, Materials Engineering **5**, 137 (1996) (in Polish).
[2] S. Deevi, V. Sikka, Intermetallics **4**, 357 (1996).
[3] M. Demura, Y. Suga, O. Umezawa, K. Kishida, E.P. George, T. Hirano, Intermetallics **9**, 157 (2001).
[4] H. Borodians'ka, M. Demura, K. Kishida, T. Hirano, Intermetallics **10**, 255 (2002).
[5] "Ni_3Al Foils Group" http://www.nims.go.jp/imc/Ni3Alfoil
[6] S.H. Kim, M.H. Oh, K. Kishida, T. Hirano, D.M. Wee, Materials Letters **58**, 2867 (2004).
[7] P. Jóźwik, Z. Bojar, Materials Engineering **3**, 753 (2006) (in Polish).
[8] Z. Bojar, P. Jóźwik, D. Zasada, Materials Engineering 22-26 (2001) (in Polish).
[9] P. Jóźwik, Doctor's Thesis, MUT, Warsaw (2004) (in Polish).
[10] E.M. Schulson, I. Baker, H.J. Frost, Material Research Society Symposium Proceedings **81**, 195 (1985).
[11] S. Hanada, S. Watanabe, W.Y. Kim, N. Masahashi, M.S. Kim, Materials Science and Engineering **A239-240**, 309 (1997).

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